## Process optimisation of encapsulated pandan (Pandanus amaryllifolius) powder using spray-drying method

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Abstract: A study on the production of spray-dried pandan (*Pandanus amaryllifolius*) powder was conducted and optimised using response surface methodology (RSM). Parameters investigated include inlet temperature  $(170-200 \,^{\circ}\text{C})$  and feed rate  $(6-12 \,\text{rpm})$ , with a preset outlet temperature of 90  $\,^{\circ}\text{C}$ . The estimated regression coefficients ( $R^2$ ) for the physicochemical characteristic and sensory responses of pandan powder were  $\geq 0.800$ , except for overall acceptability. Some mathematical models could be developed with confidence based on the results from all responses. An optimum drying process for spray drying represents conditions that would yield acceptably high colour index (such as L value, a value and b value), low moisture content, low water activity ( $a_w$ ), high solubility and high colour, flavour, odour and overall acceptability for sensory responses. Optimum conditions of 170  $\,^{\circ}\text{C}$  inlet temperature and 6 rpm feed rate, with a constant outlet temperature of 90  $\,^{\circ}\text{C}$ , were established for producing spray-dried pandan powder.

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Keywords: optimisation; Pandanus amaryllifolius; RSM; spray drying; powder

## INTRODUCTION

Spray drying is a unit operation widely used in the food industry. The most important commodity in this respect is spray-dried skim milk, but the spray drying of many other liquid foods such as fruit juices and vegetable extracts, coffee and tea extracts and other dairy products such as whey is also of economic importance.<sup>1</sup>

Spray drying is one of the most practical methods by which a solution of solids in water can be dehydrated to yield a solid final product. Other methods for the removal of water from such solutions, eg freeze-drying, are usually much more expensive, whereas a method such as liquid–liquid extraction requires the use of solvents, which is often undesirable.<sup>1</sup>

Optimisation of the dehydration process is performed to ensure rapid processing conditions yielding an acceptable-quality product and a high-throughput capacity.<sup>2</sup> Rapid food-engineering operations such as dehydration reduce the overall cost of the process; however, adverse reactions occur in biological products being dried,<sup>2,3</sup> namely loss of vitamins, development of undesirable colour, development of off-flavours, volatilisation of flavour compounds, solubility changes and loss of essential amino acids. When many factors and interactions affect desired responses, response surface methodology (RSM) is an effective tool for optimising the process.<sup>4,5</sup> The basic aim of RSM is to improve product properties via regression equations that describe relationships between input parameters and these properties.<sup>6</sup>

In Malaysia, many fruits, plants, spices and herbs contain natural flavouring and colouring compounds. The leaves of pandan (Pandanus amaryllifolius) have a strong aroma and are widely used in South East Asia as flavourings for various foods such as bakery products, sweets and even home cooking.<sup>7</sup> Today, various drying methods such as spray and drum drying are applied to produce encapsulated flavouring and colouring powders.<sup>8,9</sup> These powdered products have been widely used in ice cream, yogurt, soup, cake, tea, 'nasi lemak', a type of Malaysian traditional pandan-flavoured rice, and even Malaysian traditional coconut jam, 'seri kaya'.<sup>8</sup> Drying is an ancient process used to preserve foods. It is the most common food preservation process. Hundreds of variants are actually used in the drying of particulate solids, pastes, continuous

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Contract/grant sponsor: Ministry of Science, Technology and Environment (MOSTE); contract/grant number: IRPA 03-02-0168-EA001 (Received 22 October 2003; revised version received 28 October 2004; accepted 4 January 2005) Published online 4 May 2005

<sup>© 2005</sup> Society of Chemical Industry. J Sci Food Agric 0022-5142/2005/\$30.00

sheets, slurries or solutions; hence it provides the most diversity among food-engineering unit operations. The quality of a food powder is based on a variety of properties, depending on the specific application. In general, the final moisture content, insolubility index, rheological properties and bulk density are of primary importance.

In recent years, natural food ingredients have been recognised for their health-promoting qualities. Therefore much effort has been devoted to preparing flavouring and colouring compounds from natural sources by extraction, purification and isolation.<sup>7,10,11</sup> Continued interest in natural compounds by consumers has shown that consumers are just as concerned about their health as they are about the quality of the foods they eat.<sup>12</sup>

Nowadays the main challenges in the production of powders are the development of specific properties and the reduction of processing costs. Therefore it is important that optimum operating criteria and processing conditions be established to ensure prime quality of dried powder products during drying processes. This study describes the production of an instant pandan powder as a flavouring and colouring ingredient and evaluates the performance of the spray dryer in relation to various quality parameters of dried pandan powder by using RSM.

## MATERIALS AND METHODS Materials

Pandan (*Pandanus amaryllifolius*) leaves were the raw material used in this study. The leaves were purchased from a local farmer at Seri Kembangan market (Selangor, Malaysia). Sodium caseinate and maltodextrin DE10 were purchased from Behn Meyer (Selangor, Malaysia). Refined/bleached/deodorised (RBD) palm olein was obtained from a local retailer. The preparation of pandan powder at room temperature was carried out using the formulation shown in Table 1.<sup>13</sup>

RSM was used to optimise different combinations of parameters, namely inlet temperature and feed rate, with a preset outlet temperature of 90 °C, with respect to the physical and sensory properties of spraydried pandan powder. The RSM-based computer program Echip<sup>14</sup> was used in this study to provide the experimental design, calculate multiple regression equations and perform statistical evaluations. The different treatments of pandan powder are shown in Table 2.

Ingredient	Amount (g kg <sup>-1</sup> )		
Palm olein	240.0		
Maltodextrin DE10	136.0		
Sodium caseinate	24.0		
Pandan water extract	600.0		

 Table 2. Treatments of pandan powder

Treatment <sup>a</sup>	Preset inlet temperature (°C)	Feed rate (rpm)
1	170	9
2	200	12
3	170	9
4	180	6
5	185	12
6	200	8
7	200	12
8	170	12
9	180	10
10	170	12
11	185	12
12	190	6
13	170	6
14	200	10
15	200	6

<sup>a</sup> All treatments used a preset outlet temperature of 90 °C.

## Pandan extract and emulsion

Pandan leaves were washed thoroughly and trimmed to a smaller size (2-3 cm). The natural flavour and colour of pandan were extracted with distilled water at a ratio of 1:6 w/w (pandan leaves/distilled water) at room temperature. The composition of natural pandan was  $\sim 3\%$  (w/w). The pandan extract was stored in screw-capped bottles before being mixed at chilled temperature (9 °C).

In order to prevent the formation of lumpy particles during emulsion preparation, all ingredients except RBD palm olein were first mixed for 1 min at low speed and 2 min at high speed in a commercial blender (Waring model 32BL80, New Hartford, CT, USA). RBD palm olein (240 g) was then added to the liquid phase and mixed for 3 min at high speed. The mixture was homogenised using a laboratory homogeniser (Heidolph DIAX 900, Schwabach, Germany) with tool (Heidolph TUP 18G) at position 2 for 2 min in order to disperse the oil molecules evenly in the emulsion and reduce the risk of serum separation.

## Spray drying method

The pandan emulsion was spray dried in a pilotscale spray dryer (Mobile minor '2000' model E, Niro A/s, Soeborg, Denmark) with a feed pump (Watson Marlow type 505 S/RL, UK) at a constant outlet temperature of 90 °C. The encapsulated pandan powders were collected at the bottom of the cyclone. The samples were then stored in  $15 \text{ cm} \times 20 \text{ cm}$ aluminium packages and sealed with a sealer (Arrow type ARSH-300, Timbang dan Sukat, Selangor, Malaysia).

# Physical analysis of encapsulated pandan powder

#### Moisture content

Moisture content was determined by AACC method 44-15A.<sup>15</sup> Encapsulated pandan powder (2g) was dried at  $105 \pm 2$  °C for 24 h in an oven (Memmert

GmbH Co KG, Schwabash, Germany). Analyses were carried out in duplicate.

## Solubility

Solubility was determined by adding 5g of encapsulated powder to 100 ml of ultrahigh-temperature (UHT) recombined full cream milk at 26 °C. The mixture was agitated (50 Hz) with a magnetic stirrer (Fisher Scientific, Pittsburgh, PA, USA) at position 5. UHT recombined full cream milk was chosen for this analysis because the pandan powder is oil-based and the milk does not have to be kept refrigerated; thus it was more convenient to carry out the analysis without consideration of the milk's temperature during the test. The time required for the material to dissolve completely was recorded. Analyses were carried out in duplicate.

#### Colour

Colour was measured using a HunterLab Sphere Spectrocolorimeter (Hunter Associated Lab Inc, Reston, VA, USA). The powder was inserted into a  $3 \text{ in } \times 5$  in plastic bag before analysis. The values of L, a and b were determined. Analyses were carried out in duplicate.

#### Water activity

Water activity  $(a_w)$  was determined using an AquaLab Series 3 apparatus (Decagon Devices Inc, Pullman, WA, USA). Analyses were carried out in duplicate.

#### Sensory evaluation

Sensory evaluation was carried out by 10 trained panellists consisting of students and staff of the Faculty of Food Science and Biotechnology, Universiti Putra Malaysia using pandan-flavoured milk as the sample. The pandan-flavoured milk was prepared by adding 50 g of pandan powder to 250 ml of UHT recombined full cream milk. The ingredients were mixed for 1 min at low speed using a commercial blender (Waring model 32BL80) and then homogenised (Heidolph DIAX 900) at position 2 for 2 min. The sensory attributes evaluated were colour, flavour, odour and overall acceptability using a nine-point hedonic scale  $(1 = dislike extremely, 9 = like extremely).^{16}$  Sensory evaluations were carried out in duplicate.

#### Statistical analysis

RSM was used to investigate the optimum combination of inlet temperature ranging from 170 to 200 °C and feed rate ranging from 6 to 12 rpm, with physicochemical and sensory attributes as dependent or controllable variables. The Echip software was used to provide the experimental design, calculate equations, perform statistical evaluations and print out data. A central composite design (CCD) was used in this study. All 15 combinations are shown in Table 2. The mathematical models developed for optimisation purposes were as follows:

response = 
$$\beta_0 + \beta_1(\text{IT}) + \beta_2(\text{FR})$$
  
+  $\beta_{12}(\text{IT})(\text{FR}) + \beta_1^2(\text{IT})^2 + \beta_2^2(\text{FR})$ 

where response = score of each physicochemical property and sensory attribute evaluated,  $\beta_0$  = intercept,  $\beta_1$  = coefficient for inlet temperature in first-order term,  $\beta_2$  = coefficient for feed rate in firstorder term,  $\beta_{12}$  = coefficient for interaction between inlet temperature and feed rate,  $\beta_1^2$  = coefficient for inlet temperature in second-order term,  $\beta_2^2$  = coefficient for feed rate in second-order term, (IT) = proportional level of inlet temperature and (FR) = proportional level of feed rate.

## **RESULTS AND DISCUSSION**

The physicochemical characteristics of dried pandan powders are shown in Tables 3 and 4. The results of sensory evaluation are shown in Table 5. Generally, moisture content showed an increase with decreasing inlet temperature. Higher moisture content inhibits fat oxidation in the powder.<sup>17,18</sup> However, low  $a_w$  or low moisture content limits the growth of various microorganisms. Increasing  $a_w$  allows the growth of moulds, yeasts and bacteria.<sup>19</sup>

The spray-dried powder dissolved easily in UHT recombined full cream milk at room temperature. Generally, higher inlet temperature in addition to higher water content of the inlet feed may have resulted in denaturing more protein and hence affected solubility.<sup>10</sup>

Colour plays a very important role in the acceptance of pandan powder as an edible colouring agent. Pandan leaves are green in colour. Generally, scores for

Table 3. Moisture content, solubility and water activity of spray-dried pandan  $\mathsf{powder}^\mathsf{a}$ 

Treatment <sup>b</sup>	Moisture content (%)	Solubility (s)	Water activity
1	$2.45 \pm 0.06$	$115.3 \pm 13.4$	$0.35 \pm 0.01$
2	$1.46 \pm 0.10$	$186.5 \pm 3.1$	$0.31 \pm 0.08$
3	$2.15 \pm 0.23$	$125.5 \pm 5.0$	$0.34 \pm 0.01$
4	$1.72 \pm 0.52$	$115.0 \pm 31.1$	$0.33\pm0.00$
5	$1.70 \pm 0.28$	$120.5 \pm 31.8$	$0.40 \pm 0.03$
6	$2.74 \pm 0.26$	$197.0 \pm 7.1$	$0.29 \pm 0.17$
7	$1.52 \pm 0.08$	$159.0 \pm 8.5$	$0.30\pm0.08$
8	$2.11 \pm 0.11$	$81.0 \pm 28.3$	$0.37 \pm 0.04$
9	$1.99 \pm 0.11$	$139.5 \pm 23.3$	$0.36\pm0.01$
10	$2.03 \pm 0.17$	$90.0 \pm 21.2$	$0.39\pm0.01$
11	$1.75 \pm 0.05$	$136.5 \pm 10.6$	$0.41 \pm 0.02$
12	$1.77 \pm 0.11$	$91.5 \pm 5.7$	$0.34 \pm 0.01$
13	$1.79 \pm 0.19$	$82.5 \pm 10.6$	$0.33 \pm 0.01$
14	$1.87 \pm 0.31$	$159.5 \pm 6.4$	$0.31 \pm 0.02$
15	$2.16 \pm 0.13$	$135.5 \pm 7.8$	$0.29\pm0.03$

<sup>a</sup> Values are mean  $\pm$  SD of duplicate analyses.

<sup>b</sup> See Table 2 for treatment conditions.

Table 4. L, a and b values of spray-dried pandan powder<sup>a</sup>

Treatment <sup>b</sup>	L	а	b
1	$64.33\pm0.03$	$-9.13 \pm 0.10$	$19.56 \pm 0.25$
2	$61.77 \pm 0.12$	$-9.95 \pm 0.15$	$20.01\pm0.50$
3	$63.00\pm0.06$	$-9.24 \pm 0.07$	$19.69\pm0.11$
4	$71.62 \pm 0.10$	$-9.33\pm0.03$	$21.33\pm0.10$
5	$77.71\pm0.33$	$-10.37 \pm 0.10$	$22.42\pm0.23$
6	$68.95\pm0.32$	$-9.64\pm0.00$	$20.56\pm0.10$
7	$66.83\pm0.08$	$-10.14 \pm 0.04$	$21.51\pm0.08$
8	$74.66 \pm 0.11$	$-10.20 \pm 0.02$	$21.50\pm0.10$
9	$73.87 \pm 0.31$	$-9.76\pm0.01$	$20.36\pm0.04$
10	$74.12\pm0.12$	$-9.66 \pm 0.01$	$21.56\pm0.00$
11	$73.34\pm0.27$	$-10.45 \pm 0.29$	$22.13 \pm 0.11$
12	$77.57 \pm 0.01$	$-8.61 \pm 0.09$	$21.66 \pm 0.24$
13	$64.24\pm0.35$	$-9.33 \pm 0.01$	$20.90\pm0.12$
14	$68.26\pm0.95$	$-9.24\pm0.01$	$20.56\pm0.04$
15	$74.24\pm0.64$	$-8.14\pm0.01$	$21.68\pm0.17$

<sup>a</sup> Values are mean  $\pm$  SD of duplicate analyses.

<sup>b</sup> See Table 2 for treatment conditions.

 Table 5. Scores from sensory evaluation of pandan-flavoured UHT

 recombined full cream milk<sup>a</sup>

Treatment <sup>b</sup>	Colour	Flavour	Odour	Overall acceptability
1	5.95	5.76	6.00	5.90
2	6.25	6.40	6.15	5.85
3	6.15	5.65	5.95	5.85
4	5.75	5.90	6.20	5.90
5	6.35	6.75	6.70	6.55
6	5.55	5.05	5.15	5.60
7	6.15	6.35	6.10	5.90
8	6.15	6.35	6.10	6.30
9	6.25	5.75	6.10	5.50
10	6.05	6.15	6.15	6.10
11	6.45	5.75	5.90	6.10
12	5.05	4.40	5.55	5.05
13	6.30	5.95	5.95	5.75
14	6.05	5.75	5.75	5.75
15	4.95	4.05	5.05	4.20

<sup>a</sup> Values are mean score of 10 panellists.

<sup>b</sup> See Table 2 for treatment conditions.

colour from sensory evaluation indicated acceptance by all panellists, ranging from 4.95 to 6.35. Flavour and odour also play an important role in the acceptance of pandan powder as a flavouring agent. Generally, panellists found the flavour and odour of pandan powder in full cream milk acceptable, with scores ranging from 4.05 to 6.75 and from 5.05 to 6.70 respectively.

In all experiments the pandan powder showed a tendency to stick to the internal stainless steel surfaces of the drying chamber. This is probably due to the nature of the soluble solid. The adherence of powder to the walls of the drying chamber is a commonly recognised effect in the spray drying of solutions containing sugars and other solids, such as fruit juices and tomato products.<sup>10</sup>

Tables 6 and 7 show the estimated regression coefficients for the physical characteristic responses and sensory responses of pandan powder together with their coefficient of determination  $(R^2)$  and probability (P) of F values. It is clearly seen that, except for overall acceptability, all physical characteristic and sensory responses gave  $R^2 \ge 0.800$ . The  $R^2$  values were 0.803, 0.848, 0.891, 0.800, 0.800, 0.905, 0.894, 0.844 and 0.817 for moisture content, solubility, L value, a value, b value,  $a_w$ , colour, flavour and odour respectively. The  $R^2$  value for overall acceptability was 0.750. This means that  $R^2$  values were satisfactory and considered sufficiently accurate for prediction purposes. The closer the  $R^2$  value to unity, the better the empirical model fits the actual data.  $R^2$  values of more than 0.75 are relatively adequate for prediction purposes.20,21

A high proportion of the variability was explained by the mathematical models for moisture content, solubility, L value, a value, b value,  $a_w$ , colour, flavour, odour and overall acceptability responses of the spray-dried pandan powder, showing that the model developed for all responses appeared to be adequate.

Some models could be developed with confidence based on the results in Tables 6 and 7. For overall acceptability the *P* value obtained was less than 0.05, meaning that the model could be developed confidently (P < 0.05). The lower *P* values for moisture content, solubility, *L* value, *a* value, *b* value,  $a_w$ , colour, flavour and odour (P < 0.01), namely 0.0053, 0.0018, 0.0004, 0.0057, 0.0057, 0.0002, 0.0004, 0.0020 and 0.0039 respectively, indicated that

Table 6. Estimated regression coefficients for physical characteristic responses of pandan powder

Coefficient <sup>a</sup>	Moisture	Solubility	L	а	b	Water activity
$\beta_0$	2.11147	149.327***	72.7539	-9.60675	20.587	0.358981***
$\beta_1$	-0.000996754	2.12696	0.0659311	0.00741247***	0.0117004	-0.00158641***
$\beta_2$	-0.0284072	3.20193	0.0787014	-0.206874	0.0322801	0.00752635
$\beta_1\beta_2$	-0.00613239**	0.199164	-0.118416***	-0.0543954	-0.00943881	-0.000256525
$\beta_1^2$	0.000816706	0.0104559	-0.0292633***	0.00145305	-0.00290574	-0.000185369**
$\beta_2^2$	-0.0453191**	-3.6585*	0.326804	-0.0102811	0.141103**	0.000800711
$R^2$	0.803	0.848	0.891	0.800	0.800	0.905
Р	0.0053	0.0018	0.0004	0.0057	0.0057	0.0002

<sup>a</sup>  $\beta_0$  = intercept,  $\beta_1$  = coefficient for inlet temperatures,  $\beta_2$  = coefficient for feed rate.

\* Significant at 0.05 level, \*\* significant at 0.01 level, \*\*\* significant at 0.001 level.

Table 7. Estimated regression coefficients for sensory responses of pandan powders

Coefficienta	Colour	Flavour	Odour	Overall acceptability
$\beta_0$	6.022995**	5.51433*	5.97258**	5.8103*
$\beta_1$	-0.0164376***	-0.0242636***	-0.0173973**	-0.0208138**
β2	0.122624***	0.201184**	0.0903508	0.144737
$\beta_1 \rightarrow 2$	0.0079247	0.0117295	0.00593394	0.00602872
$\beta_1^2$	-0.000395772	2.46e-05	-0.00118897	-0.000738635
$\beta_2^2$	-0.0110758	0.0189977	0.0138269	-0.00241072
$R^2$	0.894	0.844	0.817	0.750
Р	0.0004	0.0020	0.0039	0.0151

<sup>a</sup>  $\beta_0$  = intercept,  $\beta_1$  = coefficient for inlet temperature,  $\beta_2$  = coefficient for feed rate. \* Significant at 0.05 level, \*\* significant at 0.01 level, \*\*\* significant at 0.001 level.

the models for these attributes could be developed with high confidence.

Inlet temperature was the most important factor affecting the *a* value,  $a_w$ , colour and flavour of pandan powder with high significance (P < 0.001). For odour and overall acceptability the effects were also highly significant (P < 0.01), but there was no effect on moisture content, solubility, *L* value and *b* value.

Feed rate had a highly significant (P < 0.001) effect on colour, a highly significant (P < 0.01) effect on flavour but no effect on moisture content, solubility, L value, a value, b value,  $a_w$ , odour and overall acceptability. Although there was no significant correlation with level of inlet temperature at first order, the correlation at second order was highly significant (P < 0.001) for L value.

For feed rate the correlation at second order was highly significant (P < 0.01) for moisture content and b value and significant (P < 0.05) for solubility. The interaction between inlet temperature and feed rate was highly significant (P < 0.001) for L value, highly significant (P < 0.01) for moisture content but not significant for the rest.

Contour and surface plots were generated using significant parameters for each response. An optimum drying process for spray drying represents conditions that would yield acceptably high colour index (such as L value, a value and b value), low moisture content, low  $a_w$ , high solubility and high colour, flavour, odour and overall acceptability for sensory responses. The contour plots of the responses of the independent variables show lines of constant value. Figure 1 presents the optimised response contours for the parameters of inlet temperature and feed rate.

The optimum drying conditions can then be determined by superimposing the contour plots of relevant and statistically significant responses. An optimum area is generated, and calculating the centroid of the area forms the basis of the optimum drying conditions. Optimum conditions of  $170 \,^{\circ}$ C inlet temperature and 6 rpm feed rate, with a constant outlet temperature of  $90 \,^{\circ}$ C, were established for producing spray-dried pandan powder as an edible colouring and flavouring powder.



**Figure 1.** Contour map illustrating optimum spray-drying conditions as a function of independent variables.

#### CONCLUSION

Optimum conditions for the production of encapsulated spray-dried pandan powder were established. An inlet temperature of  $170 \,^{\circ}$ C and a feed rate of 6 rpm, with a constant outlet temperature of  $90 \,^{\circ}$ C, were shown to be the most acceptable conditions to produce encapsulated pandan powder. All physicochemical characteristic and sensory responses gave  $R^2$  values relatively adequate for prediction purposes. Mathematical models could be developed with confidence based on the P values for all physicochemical characteristic and sensory responses.

#### ACKNOWLEDGEMENT

This research work was supported by the Ministry of Science, Technology and Environment (MOSTE) through Universiti Putra Malaysia (IRPA project 03-02-0168-EA001).

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